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(71) Applicant
Cabot Corporation

(Incorporated in the USA - Delaware)

950 Winter Street, P O Box 9073, Waltham, Massachusetts 02254-9073, United States of America

(72) Inventors
John E Chatfield
Donald E. Tunison III
Frederick Muller

(74) Agent and/or Address for Service
Brookes, Martin & Wilson
Prudential Buildings, 5 St Philip's Place, Birmingham, B3 2AF, United Kingdom

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(54) Continuous treatment process

(57) A process for continuously contacting a material with a bulk density of about 160.2kg/M^3(101bs./cu.ft) or less with a treating agent e.g. contacting fumed silica with polydimethylsiloxane oil in a ratio of 2:1 by weight by injecting the oil at right angles into the silica as it is being transferred in the dense phase by pipe from a storage container to a heated vessel, whereby the silica is rendered hydrophobic.

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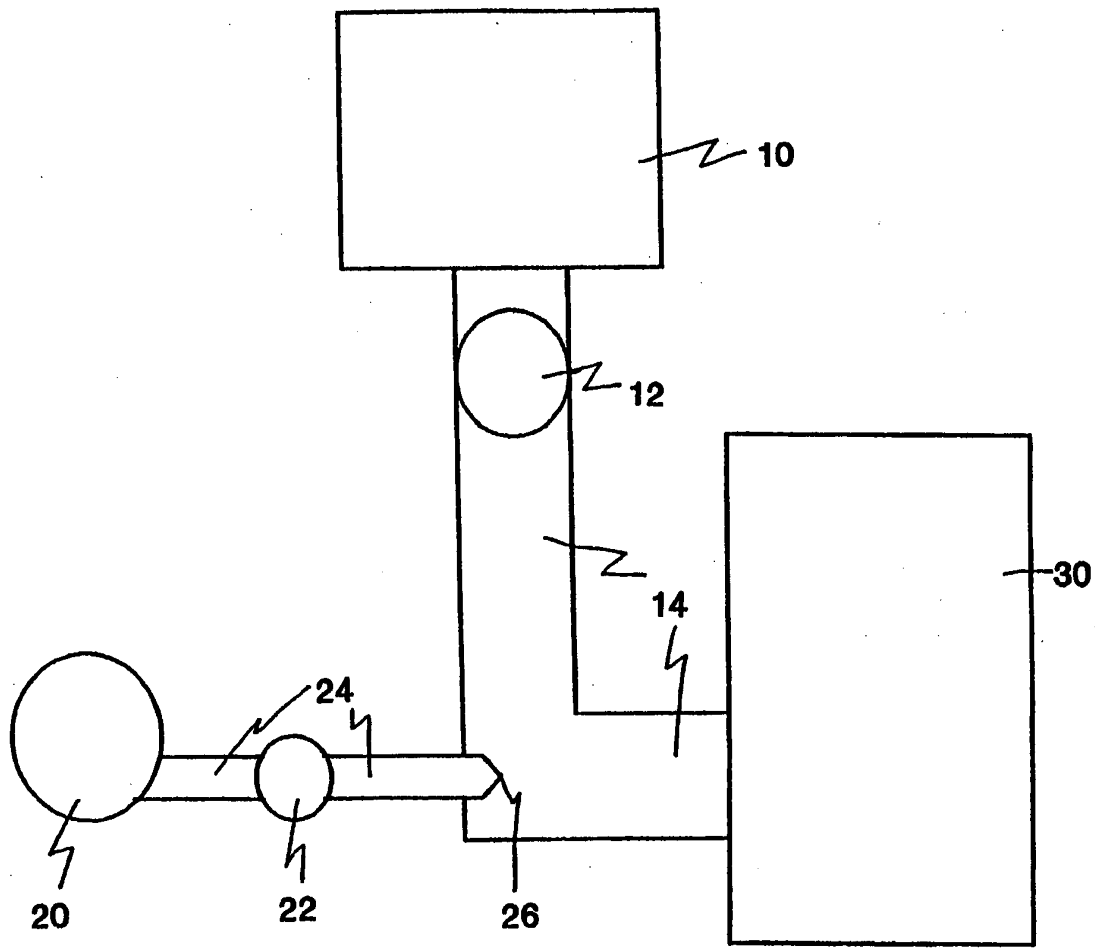

FIG. 1

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CONTINUOUS TREATMENT PROCESS

The present invention relates to a method for treating material having a bulk density of about 160.2kg/M^3 (10lbs./cu. ft.) or less with a treating agent in a continuous manner. More particularly the present invention relates to a method comprising contacting the material having a bulk density of about 160.2kg/M^3 (10lbs./cu.ft.) or less with the treating agent as the material is being transferred in dense phase into a vessel and heating the treating agent contacted material in the vessel if necessary.

Materials having a bulk density of about 160.2kg/M^3 (10lbs./cu.ft.) or less are commonly employed as fillers or reinforcing agents to improve the physical properties of compositions including rubbers, coatings, adhesives, paints and sealants. Examples of these materials include, but are not limited to, fumed silicas, precipitated silicas, fumed aluminas, carbon blacks and aerogels.

It is often desirable to treat these materials having a bulk density of 160.2kg/M^3 (10lbs./cu.ft.) or less to change their properties to render them more suitable for a particular purpose. Generally known

treating agents include, but are not limited to, liquids, vapours, atomized liquids, particulate solids etc. Generally, materials having a bulk density of about 160.2kg/M^3 (10lbs./cu.ft.) or less are treated with a treating agent in a ratio of about 1 - 5 parts, by weight, material to 1 part, by weight, treating agent.

For example, for many applications it is desirable to employ a hydrophobic filler or reinforcing agent. Thus if the material having a bulk density of about 160kg/M^3 (10lbs./cu.ft.) or less is normally hydrophilic, it must be treated to be made hydrophobic. As previously discussed, generally hydrophilic materials having a bulk density of 160.2kg/M^3 (10lbs./cu.ft.) or less are treated with treating agents to make them hydrophobic. Examples of these treating agents include, but are not limited to, silicone oils, silane oils, dimethylsiloxane oils, and hydrogen fluoride vapours.

Generally about 1 - 5 parts, by weight, of the material having a bulk density of 160.2kg/M^3 (10lbs./cu.ft.) or less are treated with 1 part, by weight, treating agent. For example, in an embodiment

of the present invention for treating fumed silica with a silane oil, about 2 - 5 parts by weight fumed silica are treated with 1 part, by weight, silane oil.

As previously discussed, one example of a material having a bulk density of 160.2kg/M^3 (10lbs./cu.ft.) or less is fumed silica. This material, also known as pyrogenic silica, is a well known reinforcing agent or filler commonly employed to improve the physical properties of compositions including silicone rubbers, coatings, adhesives and sealants. Fumed silica basically comprises finely divided silicon dioxide particles and generally has a bulk density of about 80.1kg/M^3 (5lbs./cu.ft.) or less and a surface area generally between about 50 - 400 sq.m/g.

Another example of a material having a bulk density of 160.2kg/M^3 (10lbs./cu.ft.) or less is precipitated silica. Precipitated silica is also a well known reinforcing agent or filler commonly employed to improve the physical properties of compositions including silicone rubbers, coatings, adhesives and sealants. Precipitated silica basically comprises finely divided silicon dioxide particles and

generally has a bulk density of about 160.2kg/M³ (10lbs./cu.ft.) or less. For certain applications it is desirable to employ hydrophobic fumed silicas. Generally hydrophilic fumed silica is made hydrophobic by treating the hydrophilic fumed silica with a treating agent. For example U.S. Patent No. 4,307,023 to Ettlinger et. al. discloses treating fumed silica with organosilicon compounds such as silicon oils to make the fumed silica hydrophobic and U.S. Patent No. 4,054,689 to Calvin discloses treating fumed silica with hydrogen fluoride vapours to make the fumed silica hydrophobic. Heat may be required to facilitate a reaction between the fumed silica and the treating agent.

Generally the fumed silica is treated in a batch-type process in a vessel by filling the vessel with fumed silica and then contacting the fumed silica with a treating agent. This method is disclosed in U.S. Patent No. 4,307,023. Mechanical mixing means may be employed to mix the fumed silica and the treating agent. The vessel may also be heated during this process. A similar process is also disclosed in U.S. Patent No. 4,780,108 to Razzano which discloses placing a low bulk density material in a mixing vessel and then

spraying a treating agent onto the material while it is being turbulently mixed. A batch-type method is also disclosed in U.S. Patent No. 4,054,689 although in the method disclosed in U.S. Patent No. 4,054,689 the treating agent may be added to the vessel before the fumed silica.

However these methods for treating a material having a bulk density of about 160.2kg/M^3 (10lbs./cu.ft.) or less with a treating agent by contacting the material with the treating agent in a vessel have many disadvantages. First these methods are not continuous. To maintain the desired ratio of the material having a bulk density of about 160.2kg/M^3 (10lbs./cu.ft.) or less to treating agent a fixed amount of both must be added to the vessel. Then the final product must be completely removed from the vessel and the vessel cleaned. Then the process starts over again with new quantities of material having a bulk density of about 160.2kg/M^3 (10lbs./cu.ft.) or less and treating agent.

Another disadvantage with the generally employed methods is that the means for adding the treating agent to the vessel frequently clog. For example, generally the treating agent is added to the vessel through

injection means or other openings at the end of a pipe or pipes. These injection means or openings can become clogged by the material having a bulk density of about 160.2kg/M³ (10lbs./cu.ft.) or less and therefore require frequent cleaning.

Further, in view of the fact that generally known processes are batch-type processes, there is a tendency for the treating agent to ball up in the vessel or collect on the injection means or opening. Both the injection means or opening and the vessel must therefore be cleaned to remove the treating agent balls and collected treating agent.

Also, if the vessel is heated, the treating agent balls and/or the treating agent collected on the nozzle or opening can cause fires in the vessel. These fires may also occur if excess treating agent is not thoroughly cleaned from the vessel.

Another problem with treating a material having a bulk density of about 160.2kg/M³ (10lbs./cu.ft.) or less with a treating agent by contacting the material with the treating agent in a vessel is ensuring all of the material is contacted by the treating agent. As previously discussed, mechanical mixing means are often employed to attempt to thoroughly contact all the

material having a bulk density of about 160.2kg/M^3 (10lbs./cu.ft.) or less with the treating agent. However, because of its density, a material having a bulk density of about 160.2kg/M^3 (10lbs./cu.ft.) or less is difficult to move and/or agitate by mechanical means. Therefore the material located closer to the point where the treating agent is added will tend to be contacted with too much of the treating agent. Conversely, the material located further away from the point where the treating agent is added will tend to be contacted with too little treating agent. Both the "over-contacted" material and the uncontacted material end up lowering the final product's quality.

Accordingly, one object of the present invention is to overcome the disadvantages of known methods for treating a material having a bulk density of 160.2kg/M^3 (10lbs./cu.ft.) or less with a treating agent.

Another object of the present invention is to provide a continuous method for treating a material having a bulk density of 160.2kg/M^3 (10lbs./cu.ft.) or less with a treating agent.

A further object of the present invention is to provide a method for treating a material having a bulk density of 160.2kg/M^3 (10lbs./cu.ft.) or less with a

treating agent which substantially reduces the chances of fires. A still further object of the present invention is to provide a method for treating a material having a bulk density of $160.2 kg/M^3$ (10lbs./cu.ft.) or less with a treating agent in which the material is uniformly and thoroughly contacted by the treating agent.

A still further object of the present invention is to provide a method for treating a material having a bulk density of $160.2 kg/M^3$ (10lbs./cu.ft.) or less with a treating agent which does not require frequent cleaning of the means for treating.

According to the present invention a material having a bulk density of $160.2 kg/M^3$ (10lbs./cu.ft.) or less is treated with a treating agent by contacting the material with the treating agent in a continuous manner as the material is being transferred in dense phase between a storage container and a vessel. The contacted material may be heated in the vessel if necessary.

A major advantage of the present invention is that the material having a bulk density of $160.2 kg/M^3$ (10lbs./cu.ft.) or less may be continuously treated with the treating agent.

Another advantage of the present invention is that the chance of fire in the vessel is considerably reduced.

A further advantage of the present invention is that the material having a bulk density of 160.2kg/M³ (10lbs./cu.ft.) or less is evenly and uniformly contacted by the treating agent.

A still further advantage of the present invention is that the vessel requires less frequent cleaning than in heretofore utilized treating methods.

Other advantages of the present invention will become apparent from the following more detailed description of the invention.

Figure 1 is a schematic of an embodiment of the present invention.

An embodiment of the present invention is shown in Figure 1. A material having a bulk density of 160.2kg/M³ (10lbs./cu.ft.) or less is stored in container 10 and a treating agent is stored in container 20. Pump 12 continuously pumps the material having a bulk density of 160.2kg/M³ (10lbs./cu.ft.) or less through pipe 14 in dense phase so that the bulk density of the material in the pipe is 160.2kg/M³ (10lbs./cu.ft.) or less. At the same time pump 22 is

pumping the treating agent through pipe 24, and injection means 26, into pipe 14 where the treating agent contacts the material having a bulk density of 160.2kg/M^3 (10lbs./cu.ft.) or less. If desired the treating agent may be heated in storage container 20 or pipe 24. The treating agent contacted material continues to flow through pipe 14 into vessel 30. If necessary vessel 30 may be heated to finish the treatment of the material having a bulk density of 160.,2kg/M^3 (10lbs./cu.ft.) or less with the treating agent. Injection means 26 are utilized to uniformly distribute the treating agent throughout pipe 14 and therefore to uniformly contact the material having a bulk density of 160.2kg/M^3 (10lbs./cu.ft.) or less with the treating agent. In the embodiment depicted in Figure 1 the treating agent is added in substantially the same direction as the direction of flow of the material having a bulk density of 160.2kg/M^3 (10lbs./cu.ft.) or less through pipe 14. Also in the embodiment depicted in figure 1, pipe 24 communicates with pipe 14 at an approximately 90 degree bend in pipe 14 and protrudes slightly into the interior of pipe 14 so that injection means 26 are located away from the side wall of pipe 14. As will be readily apparent to

one skilled in the art, the location of injection means 26 in pipe 14 and the location of pipe 24's communication with pipe 14 may be varied to produce optimal contact between the particular material having a bulk density of $160.2 kg/M^3$ (10lbs./cu.ft.) or less being treated and the particular treating agent being used to treat the material. Similarly, the direction in which the treating agent is added may be varied.

The present invention is well suited for treating fumed silica with a silane oil, such as polydimethylsiloxane, to make the fumed silica hydrophobic. Fumed silica is stored in container 10 and silane oil is stored in container 20. Pump 20 is a conventional diaphragm pump and pump 22 is a conventional pump. Vessel 30 is heated to a temperature sufficient to render fumed silica product hydrophobic. To begin the process pumps 20 and 22 are started. Fumed silica is thereby pumped from container 10 through pipe 14 where it is contacted by the silane oil pumped through pipe 24 and into pipe 14 through injection means 26. If desired the silane oil may be heated in either storage container 20 or pipe 24. The fumed silica and the silane oil are pumped at different rates so that the

CLAIMS

1. A process comprising;

continuously contacting a material having a bulk density of 160.2kg/M³ (10lbs./cu.ft.) or less with a treating agent while said material is being transferred in dense phase from a storage container to a vessel.

2. The process of claim 1 further comprising heating said material after said treating agent contact.

3. The process of claim 1 or 2 wherein said contacting comprises spraying said material with said treating agent.

4. The process of claim 3 wherein said material is being transferred in a pipe and wherein said spraying comprises injecting means located in the interior of said pipe.

5. The process of claim 4 wherein said spraying further comprises injecting said treating agent in substantially the same direction as the direction of travel of said material being transferred.

6. The process of claim 4 wherein said injecting means are located at an approximately 90 degree bend in said pipe.

7. The process of any one of claims 1-6 wherein the ratio, by weight, of said material to said treating agent is 1:1 to 5:1.

8. The process of any one of claims 1-7 wherein said material comprises a fumed silica.

9. The process of claim 8 wherein said treating agent comprises a silane oil.

10. The process of claim 9 wherein said silane oil comprises polydimethylsiloxane.

11. The process of claims 8, 9 or 10 wherein the ratio, by weight, of fumed silica to silane oil is 2:1 to 5:1.

12. The process of any one of claims 8-11 wherein said silane oil is heated before contacting said material.

13. The process of any one of claims 1-12 wherein said material is hydrophilic before contact with said treating agent and hydrophobic after contact with said treating agent.

14. A process comprising;

continuously contacting a material having a bulk density of 160.2kg/M^3 (10lbs./cu.ft.) or less with a liquid while said material is being transferred in dense phase from a storage container to a vessel.

15. The process of claim 14 wherein said material is hydrophilic before contact with said liquid and hydrophobic after contact with said liquid.

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